INFORMATION ACQUISITION APPARATUS, CROSS SECTION EVALUATING APPARATUS, CROSS SECTION EVALUATING METHOD, AND CROSS SECTION WORKING APPARATUS

This application is a continuation-in-part of Application No. 10/488,974 filed on March 9, 2004, which is the National Stage of International Application No. PCT/JP02/10277, filed on October 2, 2002.

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to an information acquisition apparatus for acquiring information on a specimen, and more particularly to a cross section evaluating apparatus and a cross section evaluating method for evaluating the cross section of a specimen of which state and shape vary according to a change in temperature 及び断面加工装置に関する.

Related Background Art

The demand for evaluation of a cross section or formation of a fine structure in organic materials, including bio-origin materials and plastics, is increasing together with the recent increase of functional devices.

As the principal methods of preparing a cross section, utilized for obtaining information on the

structure of an organic material, there are known, for example, a cutting method with a blade, an embedding method in resin, an embedding method by freezing, a breaking method by freezing, an ion etching method etc., but, in case of observing the internal structure of an organic material with an optical microscope, there is usually adopted a method of embedding the organic material in a resin and cutting it with a microtome.

However, the observation with the optical microscope is limited to a macroscopic analysis of the cross section, and, since the cut-out position cannot be designated, a large amount of work has been necessary in repeating the cross-section preparing operation, in order to achieve observation and analysis of the structure of the designated position.

For this reason, there has recently been developed an FIB-SEM apparatus in which a working function by an FIB (focused ion beam) apparatus is attached to an SEM (scanning electron microscope). The FIB apparatus irradiates a working specimen with a finely focused ion beam from an ion source, thereby achieving a working operation such as etching. The etching technology with such FIB apparatus is becoming more and more popular, and is currently widely employed for a structural analysis and a defect analysis of a semiconductor material or the

like, and for preparing a specimen for a transmission electron microscope. The FIB-SEM apparatus is capable of executing a step of etching a specimen and a step of observing the cross section of the specimen by the SEM within a single apparatus, thus being capable of designating a cut-out position and observing and analyzing the structure in such designated position.

Such FIB-SEM apparatus has been proposed in various configurations. For example, the Japanese Patent Application Laid-Open No. H01-181529 proposes an apparatus capable, while the specimen is fixed, of SEM observation of the working depth in the course of FIB working and SIM (scanning ion microscope) observation of the surface of the specimen in the course of working. This apparatus is so constructed that a focused ion beam (FIB) from an FIB generation unit and an electron beam from an electron beam generation unit irradiates, with respectively different angles, a same position of the fixed specimen, and the working by the FIB and the SEM (or SIM) observation by detecting secondary electrons emitted from the specimen in response to the irradiation with the electron beam (or FIB) are alternately executed, whereby the working state of the specimen can be monitored in the course of the working process.

In addition, the Japanese Patent Application Laid-Open No. H09-274883 proposes a configuration of irradiating an electrode with a beam to prevent charging of the specimen in the course of FIB working, thereby enabling a highly precise working.

DISCLOSURE OF THE INVENTION

However, in case the aforementioned conventional FIB-SEM apparatus is used for observation and analysis of the cross-sectional structure of a specimen of which state or shape changes by the temperature such as an organic material, the heat generated in the course of FIB working causes a change in the temperature of the specimen, thereby varying the state or shape thereof, whereby the cross-sectional structure of the specimen cannot be exactly analyzed.

In consideration of the foregoing, an object of the present invention is to provide an information acquisition apparatus capable of resolving the aforementioned drawbacks and acquiring the information on the surface of which information is desired, in a state where the temperature of the specimen is regulated.

Another object of the present invention is to provide a cross section evaluating apparatus and a cross section evaluating method capable of resolving

the aforementioned drawbacks and analyzing the cross section in a state where the temperature of the specimen is regulated.

Still another object of the present invention is to provide a working apparatus, a work portion evaluating apparatus and a working method, capable of resolving the aforementioned drawbacks, and of working a specimen and exactly acquiring the information of the work portion in a state where the temperature of the specimen is regulated.

The above-mentioned objects can be attained, according to the present invention, by an information acquisition apparatus comprising a stage for placing a specimen, a temperature regulation means for regulating the temperature of the specimen, an exposure means for exposing a surface of the specimen of which surface information is desired, and an information acquisition means for acquiring the information relating to the surface exposed by the exposure means.

According to the present invention, there is also provided a cross section evaluating apparatus comprising a stage for placing a specimen, a temperature regulation means for regulating the temperature of the specimen, an ion beam generation means for irradiating the specimen with an ion beam thereby cutting out a cross section or working the

specimen, an electron beam generation means for irradiating the specimen with an electron beam, and a detection means for detecting an emission signal emitted from the specimen in response to the irradiation with the ion beam or the irradiation with the electron beam, to acquire information from the detection means is acquired.

There is also provided a cross section evaluating apparatus provided with the aforementioned cross section evaluating apparatus further comprising an information acquisition means for irradiating a predetermined portion of the specimen with the ion beam to cut out a cross section or work the specimen, scanning the surface of the predetermined portion or the cut-out cross section with the ion beam or the electron beam, and acquiring an image information relating to the surface of the predetermined portion or the cut-out cross section based on emission signals from plural point detected by the detection means in synchronization with the scanning.

According to the present invention, there is also provided a cross section evaluating method comprising the steps of regulating the temperature of a specimen, irradiating a predetermined portion of the specimen with an ion beam to cut out a cross section, and scanning the cut-out cross section with an electron beam and acquiring an image relating to

the cross section from an emission signal emitted from plural points in synchronization with the scanning.

According to the present invention as described in the foregoing, the specimen is always subjected to temperature regulation, so that the specimen is always maintained at a desired temperature even in the course of FIB working and is therefore prevented from changes in the state or shape as encountered in the conventional technologies.

また、本発明によれば、

試料の断面を加工するための装置であって、

該試料を載置するための載置台と、

該試料の温度を調整するための温度調整手段と、

該試料に対してビームを照射して該試料の加工を行うためのビーム発生手段と、

加工前に該載置台と該試料を搬送する前に該試料と該載置台を収納して密封するための密封手段と、を具備していることを特徴とする断面加工装置が提供される。

本発明においては、前記温度調整手段により、前記試料を予め設定された温度に調整した状態で、前記ビーム発生手段による試料加工、及び検出手段による情報の取得、大気保護手段付カバーを取り付け後にガス導入手段のついたカバー内にガス導入を行うことができる。

また、本発明においては、前記温度調整手段は、前記試料を室温以下の温度に冷却する冷却手段を具備している構成とすることができる。

また、本発明においては、前記載置台、前記ビーム発生手段及び検出手段は、雰囲気制御可能なチャンバー内に配置され、該チャンバー内に残留するガスを捕捉するトラップ手段を更に具備している構成とすることができる。

また、本発明によれば、

試料の温度を調整する第１の工程と、

該試料にビームを照射して断面の切り出しを行う第２の工程と、

該温度制御された試料を密封する第３の工程と、

該密封された試料を他の装置へ搬送する第４の工程と、

該搬送された試料を前記他の装置を用いて評価を行う第５の工程と、

を有することを特徴とする断面評価方法が提供される。

本発明においては、前記密封する試料の周囲にガスを導入する工程を更に行うこともできる。

また、本発明においては、前記導入ガスが、不活性ガスあるいはドライ窒素の前記試料にダメージを与えないガスとすることができる。

In the present invention, a cross section not only indicates a plane inside the specimen seen from a point, but also, even in case the specimen is subjected to a working (including deposition or etching), a plane observable seen from a view point after such working.

Also according to the present invention, even in a specimen showing a change in the state or shape by a temperature change, the exposure of a surface of which information is desired and the acquisition of information are executed in a state where the temperature of such specimen is regulated, so that exact information can be acquired from the surface of which information is desired.

Also in case the present invention is applied to a cross section evaluating apparatus, there can be executed a working of the cross section, an

observation (SEM or SIM observation) and an elementary analysis can be executed while a specimen, showing a change in the state or shape by a temperature change, is maintained at a desired temperature, so that there can achieved an exact morphological analysis of a micro cross section of the specimen can be executed.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a view schematically showing the configuration of a scanning electron microscope for cross sectional observation, constituting a first embodiment of the cross section evaluating apparatus of the present invention;

Fig. 2 is a block diagram schematically showing the configuration of a specimen stage with a temperature controller, constituting an example of a temperature holding unit shown in Fig. 1;

Fig. 3 is a flow chart showing a procedure of cross sectional evaluation, utilizing the scanning electron microscope for cross sectional observation shown in Fig. 1;

Fig. 4 is a view schematically showing the configuration of a scanning electron microscope for cross sectional observation, constituting a second embodiment of the cross section evaluating apparatus of the present invention;

Fig. 5 is a block diagram schematically showing the configuration of a specimen stage with a temperature controller, constituting an example of a temperature holding unit shown in Fig. 4;

Fig. 6 is a view schematically showing the configuration of a scanning electron microscope for cross sectional observation, constituting a third embodiment of the cross section evaluating apparatus of the present invention;

Fig. 7 is a view schematically showing the configuration of a scanning electron microscope for cross sectional observation, constituting a fourth embodiment of the cross section evaluating apparatus of the present invention;

Fig. 8A is a schematic view showing an example of a cross section prepared by an FIB working, while Fig. 8B is a schematic view showing a state of SEM observation of the cross section shown in Fig. 8A; and

Fig. 9A is a schematic view showing an example of a cross section prepared by an FIB working, while Fig. 9B is a schematic view showing a state of elementary analysis of the cross section shown in Fig. 9A.

【図１０】は、本発明の断面評価装置の第６の実施形態である、断面加工用集束イオンビーム加工装置の概略構成図である。

【図１１】は、図１０に示す断面加工用集束イオンビーム加工装置を用いた試料

の断面評価の一手順を示すフローチャート図である。

【図１２】は、本発明の断面評価装置の第７の実施形態である、断面加工用集束イオンビーム加工装置の概略構成図である。

【図１３】本発明の断面評価装置の第８の実施形態である、加工面観察用走査型電子顕微鏡の概略構成図である。

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Now the present invention will be clarified in detail by embodiments thereof, with reference to the accompanying drawings.

(Embodiment 1)

Fig. 1 schematically shows the configuration of a scanning electron microscope for cross sectional observation, constituting a first embodiment of the cross section evaluating apparatus of the present invention. The electron microscope is provided with a temperature holding unit 2, on which a specimen 1 is fixed and which maintains the temperature of the specimen at a preset temperature. Temperature holding unit 2 can be accommodated in a specimen chamber 3.

Specimen chamber 3 is provided with an ion beam generation unit 4 for irradiating specimen 1, fixed to temperature holding unit 2 with an ion beam, and an electron beam generation unit 5 for irradiating the specimen with an electron beam and also with an electron detector 6 for detecting secondary electrons

emitted from specimen 1 by the irradiation with the electron beam or the ion beam. The interior of specimen chamber 3 can be evacuated by a pump unrepresented in the figure to hold a predetermined low pressure, whereby the irradiation with the ion beam or the electron beam is rendered possible. In the present invention, the interior of the specimen chamber is preferably maintained at a pressure of 1×10^{-10} Pa to 1×10^{-2} Pa.

Ion beam generation unit 4 is used for irradiating specimen 1 with the ion beam thereby cutting out a cross section, and it can also be used for SIM observation. In case of SIM observation, secondary electrons generated when specimen 1 is irradiated with the ion beam are detected by electron detector 6, and an image is formed based on a detection signal from electron detector 6.

Electron beam generation unit 5 is used for SEM observation. In case of SEM observation, the secondary electrons generated when specimen 1 is irradiated with the electron beam are detected by electron detector 6, and an image is formed based on a detection signal from electron detector 6.

The detection signal from electron detector 6 is supplied to a control unit 7, which executes image formations in the aforementioned SIM and SEM observations. For example, control unit 7 acquires

image information (mapping information) from the detection signal supplied from electron detector 6, and forms an image by causing an unrepresented display apparatus to display such image information. In addition, control unit 7 controls the ion beam generation in ion beam generation unit 4 and the electron beam generation in electron beam generation unit 5, and controls the irradiation and scanning of the ion beam and the electron beam onto specimen 1. The beam scanning operation can be controlled in the beam side and/or in the stage side on which the specimen is fixed, but the control at the beam side is preferable in consideration of the scanning speed etc. Also the irradiating positions of the ion beam and the electron beam can be respectively so controlled that they mutually coincide on specimen 1.

The electron beam generation unit and the ion beam generation unit may be so constructed as disclosed in Japanese Patent Application Laid-Open Nos. H11-260307 and H01-181529.

(Configuration of temperature regulating means)

Temperature regulating means in the present embodiment is provided with a temperature holding unit capable of regulating temperature of the specimen.

Temperature holding unit 2 is, for example, comprised of a specimen stage having a temperature

controller. Fig. 2 schematically shows the configuration of the specimen stage with temperature controller.

Referring to Fig. 2, the specimen stage with temperature controller is comprised of a specimen stage 8 having a temperature varying mechanism 10 in a portion where specimen 1 is fixed, a thermometer 9a for directly detecting the temperature of specimen 1, a thermometer 9b mounting in a part of temperature varying mechanism 10 for detecting the temperature in the vicinity of specimen 1, and a temperature control unit 7a for regulating the temperature of temperature varying mechanism 10 based on the temperature detected by thermometer 9b to keep the temperature of specimen 1 at a preset temperature.

Though not represented in Fig. 2, there is also provided a display unit for displaying the temperature detected by thermometer 9a, whereby the operator can confirm the temperature of specimen 1, based on the temperature displayed on the display unit. Temperature control unit 7a may also be so constructed as to regulate the temperature in temperature varying mechanism 10 based on the temperatures detected by both the thermometers 9a and 9b, thereby controlling the temperature of specimen 1 in more precise manner.

Temperature varying mechanism 10 is constructed

as a unit together with thermometer 9b, whereby a unit capable of control in a required temperature range can be installed in specimen stage 8. Such unit can be, for example, a high temperature unit having a heating mechanism such as a heater, or a low temperature having a cooling mechanism. Also, if necessary, there may be used a unit provided with a temperature varying function relating to both a lower temperature region than the room temperature and a higher temperature than the room temperature region of the room temperature.

Specimen stage 8 is capable of mechanically move specimen 1 in the vertical or horizontal direction, or rotate or incline specimen 1, thereby shifting specimen 1 to a desired position of evaluation. The movement control of specimen 1 by specimen stage 8 is conducted by the aforementioned control unit 7.

The aforementioned cooling mechanism can be comprised of a set of for example a Peltier element or a helium freezing device. Otherwise there may be adopted a system of providing a coolant pipe for flowing a cooling medium in a side of the temperature holding unit opposed to the specimen fixing portion to maintain a cooling medium such as liquid nitrogen and water in thermal contact with the temperature holding unit.

Also in order to increase the absorption

efficiency for the heat generated in the course of working, there is preferably adopted a measure for improving the contact efficiency between the specimen and the cooling unit (temperature holding unit).

Such measure can be, for example, the use of a specimen holder which is so constructed as to wrap around the specimen but not to intercept the optical system of the apparatus to be used in the working and observing operations, or working the specimen in a shape matching the shape of the stage and supporting the specimen with a maximum contact area on the stage.

It is also possible to provide a cooling member which covers only a non-worked area of the specimen so as not to intercept the beam systems.

(Evaluating method for cross section of specimen)

In the following there will be explained a cross section evaluating method of the present invention.

Fig. 3 is a flow chart showing a sequence of cross sectional evaluation of a specimen with the scanning electron microscope for cross sectional observation shown in Fig. 1. In the following there will be given an explanation on the procedure of cross sectional observation, with reference to Fig. 3, together with a detailed explanation on the control for the SEM and SIM observations by control unit 7 and on the temperature control on the specimen by temperature control unit 7a with such procedure.

At first specimen 1 is fixed on a predetermined position (temperature varying mechanism 10) of specimen stage 8 (step S10) and inserted in specimen chamber 3, and an evaluation temperature is set (step S11). In response to the setting of the evaluation temperature, temperature control unit 7a controls temperature in temperature varying mechanism 10 whereby the temperature is kept at the set evaluation temperature. In this state, the temperature of specimen 1 is detected by thermometer 9a, and the operator can confirm whether specimen 1 is maintained at the evaluation temperature based on the detection temperature displayed on the unrepresented display unit.

In the present embodiment, it is preferable to effect the working in a state where the specimen is cooled from the room temperature. Also a cooling to lower than 0°C is more preferable because the specimen can be solidified if it contains moisture.

In such a cooling process, it is preferred to cool at first the specimen to a predetermined temperature lower than the room temperature, then hold the specimen in a reduced pressure and execute a working operation by the irradiation of a focused beam while absorbing the heat generated from the vicinity of the irradiated portion of the specimen to retain the shape of the non-irradiated portion.

Also the cooling of the specimen may be achieved by rapid cooling from the room temperature. In such a case, a cooling rate of 40°C/min or higher is preferred. This method allows to observe the cross section in a rapidly cooled state in case of measuring the cross sectional state of a mixture of which dispersion state varies depending on the temperature.

The cooling step is preferably executed before the pressure reducing step, thereby allowing to suppress the evaporation of the specimen caused by the reduced pressure. However, if the specimen consists of a substance showing little evaporation, the cooling may be executed simultaneously with the pressure reduction.

The cooling depends on the specimen to be processed. In case of an ordinary organic material such as PET, it is preferably cooled to a temperature range of -0 to -200°C, preferably -50 to -150°C.

Also if the working time or the cooling time becomes excessively long at the cooling to the low temperature, a remaining gas in the specimen chamber or the substance generated at the working may be adsorbed in the specimen of low temperature, thereby eventually hindering the desired working or observation. It is therefore preferable to provide trap means for absorbing the remaining gas or the

substance generated at the working operation and to execute the working or the acquisition of information while cooling such trap means.

The method of the present invention is advantageously applicable in case the object specimen is an organic material, particularly a material susceptible to heat such as a protein or other biological substances, or a moisture-containing composition. It is particularly preferable for a composition containing moisture, since the working can be executed while the moisture is retained in the specimen.

In particular, the irradiation with the focused ion beam is executed under a reduced pressure. Therefore, in case of working on a composition containing moisture or organic molecules of high volatility, there may result evaporation of moisture or such molecules by the heat generated in the course of the working operation, and the presence of the temperature regulating means of the present invention is highly effective.

It is also preferable, in order to achieve more exact working and structural evaluation, to provide a step of determining in advance an appropriate holding temperature at the working. Such preferred holding temperature can be determined by employing a specimen, equivalent to the specimen to be worked, as a

reference, executing the working operation at plural temperatures and investigating the relationship between the damage in the worked portion and the cooling temperature.

In an ordinary FIB working apparatus, it has been customary to move the specimen, after the working thereof, to an SEM or another apparatus for executing operation etc., but the move to the observation means in the temperature controlled state has been difficult. The present embodiment provides a working apparatus capable of working and observation on the specimen in a cooled state, without influence on the worked surface for example by the deposition of water drops on the specimen at the cooling.

After the confirmation that specimen 1 is maintained at the evaluation temperature, there is executed SEM observation of the surface of specimen 1, under constant confirmation of the temperature thereof (step S12). In the SEM observation, control unit 7 controls the electron beam irradiation by electron beam generation unit 5 and the movement of specimen stage 8, whereby specimen 1 is scanned by the electron beam from electron beam generation unit 5. In synchronization with the scanning operation, electron detector 6 detects the secondary electrons, and control unit 7 displays an SEM image, based on

the detection signal of the secondary electrons, on the unrepresented display unit. Thus, the operator can execute SEM observation of the surface of specimen 1.

Subsequently, based on the image obtained by the SEM observation of the surface of specimen 1 (SEM image displayed on the display unit), the cross section position to be evaluated is precisely determined (step S13), and thus determined cross section position to be evaluated is further subjected to an SIM observation (step S14). In the SIM observation, control unit 7 controls the ion beam irradiation by ion beam generation unit 4 and the movement of specimen stage 8, whereby specimen 1 is scanned in the range of the cross section position to be evaluated by the ion beam from ion beam generation unit 4. In synchronization with the scanning operation, electron detector 6 detects the secondary electrons, and control unit 7 displays an SIM image, based on the detection signal of the secondary electrons, on the unrepresented display unit. Thus, the operator can execute SIM observation of the surface of specimen 1 at the cross section position to be evaluated determined in the step S14.

Then there are set FIB working conditions (step S15). In this setting of the FIB working conditions, a cut-out area and a cut-out position are determined

on the SIM image obtained by the SIM observation of the surface in the step S14, and there are set the cross section working conditions including an acceleration voltage, a beam current and a beam diameter. The cross section working conditions include crude working conditions and finish working conditions, which are both set at this point. In the crude working conditions, the beam diameter and the working energy are larger than those in the finish working conditions. The cut-out area and the cut-out position can be determined on the SEM image obtained in the foregoing step S14, but, in consideration of the precision, they are preferably determined on the SIM image obtained with the ion beam which is used in the actual working.

After the setting of the FIB working conditions, there is executed an FIB working (crude working) (step S16). In the crude working, control unit 7 controls the ion beam generation unit 7 according to the crude working conditions set as explained in the foregoing, and also controls the movement of specimen stage 8 whereby the cut-out area and cut-out position determined in the step S15 is irradiated with the ion beam of an amount necessary for cutting.

After the crude working, the surface of specimen 1 is subjected to an SIM observation to confirm, on an image obtained by such SIM observation (SIM image),

whether the working has proceeded close to the desired position (step S17). Also the cross section prepared by the crude working is subjected to an SEM observation to confirm the state (coarseness) of the cross section (step S18). In case the working has not proceeded close to the desired position, the aforementioned steps S16 and S17 are repeated. The steps S16 and S17 are repeated also in case the worked cross section is extremely coarse, but, in such case, there is added, for example, an operation of gradually reducing the amount of ion beam. The SIM observation of the surface in the step S17 is similarly controlled as in the foregoing step S12. Also the SEM observation of the cross section in the step S18 is controlled basically similar to the aforementioned step S12, except that specimen stage 8 is so moved that the worked cross section is irradiated by the electron beam. In this operation, the electron beam may have any incident angle to the cross section as long as an SEM image can be obtained.

After the confirmation that the crude working has proceeded close to the desired position, there is executed an FIB working (finish working) (step S19). In the finish working, control unit 7 controls the ion beam generation unit 7 according to the finish working conditions set as explained in the foregoing, and also controls the movement of specimen stage 8

whereby the crude finished portion obtained in the step S16 is irradiated with the ion beam of an amount necessary for finish working. Such finish working allows to obtain a smooth cross section, for example, enabling the observation with a high magnification with the scanning electron microscope.

Finally, thus prepared cross section of specimen 1 is subjected to an SEM observation (step S20). The control in such cross sectional SEM observation is same as that in the foregoing step S18.

As explained in the foregoing, the scanning electron microscope for cross sectional observation of the present embodiment is capable of maintaining the evaluated specimen 1 always at the set temperature, so that the state and morphology of specimen 1 do not change in the course of the FIB working. Consequently the fine structural analysis can be achieved in precise manner.

Also, the temperature of the specimen, selected in the working operation with the ion beam is preferably same as that selected at the observing operation, but the temperature in the working operation may be selected lower than that in the observing operation. In such case, there may be a temperature difference of 10 to 50°C between the working process and the observation process.

(Embodiment 2)

Fig. 4 schematically shows the configuration of a scanning electron microscope for cross sectional observation, constituting a second embodiment of the cross section evaluating apparatus of the present invention. This electron microscope is substantially same in configuration as that of the first embodiment, except for the presence of an X-ray detector 11 for detecting characteristic X-rays emitted from specimen 1 in response to the electron beam irradiation. In Fig. 4, components equivalent to those shown in the foregoing are represented by like numbers.

Control unit 7 receives a detection signal from the X-ray detector 11, and, by scanning specimen 1 with the electron beam from electron beam generation unit 5, can execute an elementary analysis in the scanned range. Thus, the present embodiment is capable of an elementary analysis, in addition to the SEM observation and the SIM observation.

The electron microscope of the present embodiment is capable, in addition to the cross sectional evaluation of the specimen by the procedure shown in Fig. 3, of a cross sectional evaluation by the elementary analysis utilizing the aforementioned X-ray detector 11. More specifically, the elementary analysis utilizing the X-ray detector 11 is executed instead of the cross sectional SEM observation (or parallel thereto) in the step S20 in the evaluation

procedure shown in Fig. 3. In the elementary analysis, control unit 7 controls the movement of specimen stage 8 in such a manner that the prepared cross section is irradiated by the electron beam from electron beam generation unit 5, and scans the cross section with the electron beam. In synchronization with the scanning operation, the X-ray detector 11 detects the characteristic X-rays from plural measuring points, and control unit 7 displays mapping information, based on the detection signal of thereof, on the unrepresented display unit. Otherwise, after the scanning of the cross section with the electron beam, a necessary position is irradiated with the electron beam and the elementary analysis is executed by detecting the characteristic X-rays generated from the irradiated position.

In order to improve the precision of the elementary analysis utilizing the aforementioned X-ray detector 11, a specimen stage with a temperature controller as shown in Fig. 5 may be employed as temperature holding unit 2. This specimen stage with temperature controller is same in configuration as that shown in Fig. 2, except for the position of temperature varying mechanism 10 and the fixing position for specimen 1. In the configuration shown in Fig. 5, temperature varying mechanism 10 is so provided that a lateral face 10a thereof is

positioned at an edge portion 8a of specimen stage 8, whereby the working of cross section can be directly executed on a lateral face 1a of specimen 1 fixed on temperature varying mechanism 10.

Thus, by employing such specimen stage with temperature controller as explained above, it is rendered possible to irradiate a right-hand portion (lateral face 1a) of specimen 1 with the ion beam thereby forming a cross section in this portion. Such formation of the cross section at the side of the lateral face 1a of specimen 1 allows to position the cross section closer to the X-ray detector 11, and the precision of the elementary analysis can be improved by such positioning of the cross section closer to the X-ray detector 11. Also by inclining the specimen stage toward the detector, it is possible to improve the detection efficiency of the generated characteristic X-rays, and to further improve the precision of the elementary analysis.

Also such working of the cross section allows to position the cross section closer to electron beam generation unit 5 whereby the precision of the SEM image obtained with electron detector 6 can also be improved.

In the embodiments explained in the foregoing, the working of the specimen with the ion beam does not involve generation of a shear stress, a

compression stress or a tensile stress as encountered in the mechanical working method such as cutting or grinding, so that a sharp cross section can be prepared even in a composite specimen consisting of a mixture of materials different in hardness or brittleness, a specimen including voids, a fine structure of organic materials formed on a substrate, a specimen easily soluble in a solvent etc.

Also, since the specimen can be maintained at the set temperature, it is possible to directly observe the designated position without destructing the layer structure, even in a specimen including a material which changes the state or shape by the temperature.

The cross section evaluating method in the foregoing embodiments is effective for analyzing, at a desired temperature, a polymer structure on various substrates such as glass, a polymer structure containing micro particles or liquid crystals, a structure of particle dispersion in a fibrous material, or a specimen containing a material showing a temperature-dependent transition. It is naturally effective also for a material which is easily damaged by an ion beam or an electron beam.

The foregoing embodiments have been explained by an apparatus for executing the SEM observation, SIM observation and elementary analysis, but the present

invention is not limited to such embodiments and is applicable also to an apparatus for executing various analyses such as mass analysis.

Further, the specimen stage with temperature controller shown in Fig. 5 can also be used as temperature holding unit 2 of the scanning electron microscope for cross sectional observation shown in Fig. 1.

(Embodiment 3)

In addition to the configurations of the foregoing embodiments 1 and 2, there may be provided a reactive gas introducing pipe 13 as shown in Fig. 6, in the vicinity of the specimen stage, thereby introducing a reactive gas to the vicinity of the specimen in the course of the FIB working. There are also shown a valve 14 and a gas source container 15.

In such case, there can be executed ion beam-assisted gas etching or gas deposition depending on the selected conditions of ion beam, gas and temperature, thereby working the surface of the specimen into an arbitrary shape. The observation (SEM observation or SIM observation) of thus worked surface allows to obtain exact information on the surface thus worked into the desired shape.

The gas introducing aperture is so three-dimensionally positioned as not to obstruct the detector or the beam system.

A well-known example of FIB-assisted deposition is tungsten deposition utilizing hexacarbonyl tungsten ($W(CO)_6$) and Ga-FIB.

Also it is possible to blow an organometallic gas around the FIB irradiating point, thereby causing a reaction between the FIB and the gas to deposit the metal of the gas onto the substrate.

A conventional FIB-assisted deposition apparatus without the cooling mechanism has been associated with a drawback that the underlying material is removed by the FIB before the FIB-assisted deposition is started. Therefore, the present invention is advantageous as a method of forming a desired inorganic material.

It is also possible blow an etching gas around the FIB irradiating point, thereby inducing a reactive etching locally in the beam irradiating position, and enabling a micro working of a high speed and a high selectivity.

The aforementioned FIB-assisted etching and FIB-assisted deposition can be executed under the conditions as described in Japanese Patent Application Laid-Open No. H07-312196.

(Embodiment 4)

As shown in Fig. 7, the present embodiment is provided, in addition to the configuration of the embodiment 1, with trap means 16 for preventing re-

deposition of the gas remaining in the specimen chamber or the substances generated at the working operation, onto the specimen. Such trap means is composed for example of a material of high thermal conductivity such as a metal, and is maintained at a temperature equal to or lower than that of the specimen while it is cooled.

The present embodiment is effective, in case of working or observation in a state of maintaining the specimen lower than the room temperature, in preventing the deposition of impurities onto the specimen. For example, in the aforementioned FIB-assisted deposition, there may be formed an impurity layer between the deposition layer and the worked specimen, thereby hindering to achieve the desired function.

Such trap means is provided, in a state where the stage with the specimen supported thereon, the ion beam generation means, the electron beam generation means and the detection means are positioned, in such a position as not to hinder the beam systems in the detecting or working operation. For improving the trapping efficiency, such trap means is preferably positioned as close as possible to the specimen, as long as it does not hinder such detecting or working operation. Also the trap means may be provided in more than one unit in the specimen

chamber maintained at a low pressure.

(Embodiment 5)

The present embodiment shows an example of applying the apparatus of the present invention as a cross section evaluating apparatus in a manufacturing process for a liquid crystal display device or an organic semiconductor device.

In the present embodiment, there will be explained a case of executing temperature regulation on the specimen of a relatively large area.

In case of exactly evaluating the cross sectional state in a part of a large-sized specimen, such as a glass substrate coated with liquid crystal and to be used in a large-size liquid crystal display device, it is preferable to regulate the temperature of the entire substrate, though a local temperature regulation of an area around the worked portion is also possible. In such case, the entire holder may be cooled by providing a coolant pipe for circulating a cooling medium, in a position opposed to the specimen supporting surface of the temperature holding unit.

（実施形態６）

図１０は、本発明の断面加工装置の第６の実施形態である、断面加工用集束イオンビーム装置の概略構成図である。集束イオンビーム装置は、試料１が固定されるとともに固定された試料１の温度を設定された温度に保つ保温部２ａならびに保温部２ａを支える保持台２を備える。この保温部２ａは、試料室３内に収容可能である。

試料室３には、保温部２ａに固定された試料１に対してイオンビームを照射するイオンビーム発生部４及びイオンビームの照射によって試料１から発生する信号を検出する検出部５が設けられており、更にガス導入部６、カバー７が設けられている。試料室３内は、不図示のポンプによって排気され、所定の低圧力を保てるようになっており、これによりイオンビームの照射が可能となっている。本発明においては、試料室内の圧力を1×10^{2}Ｐａ以下にすることが好ましい。

イオンビーム発生部４は、試料１にイオンビームを照射して断面を切り出すために用いられる他、ＳＩＭ観察のために用いることも可能である。ＳＩＭ観察の場合には、試料１にイオンビームを照射したときに発生する２次電子又は２次イオンが検出器５にて検出され、検出器５からの検出信号に基づいて映像化が行われる。

ガス導入部６は、試料１周辺の雰囲気を制御するために用いられる。また、試料室３内の圧力を上昇させるために用いることもできる。不図示のシャッターによってイオンビーム発生部４と検出器５を高真空に保ったまま試料室側と真空ラインを切り離した後、試料室３内のカバー７を保温部２ａごと試料１に被せることができ、試料室３をリークする。試料１は、カバー７内にあり、試料周辺の雰囲気は、ガス導入部６から制御して行われるため、試料１の材料、温度によって、必要な条件のものを選択可能である。また、試料室３のリークは、不図示のリーク弁から行うことも可能である。

検出器５からの検出信号は制御部９に供給されており、上記のＳＩＭ観察時の映像化及びＳＥＭ観察時の映像化はこの制御部９によって行われる。例えば、制御部９は、検出器５からの検出信号から映像情報（マッピング情報）を取得し、この取得した映像情報を不図示の表示装置に表示させることで映像化を行う。この他、制御部９は、イオンビーム発生部４におけるイオンビームの発生を制御したり、それらイオンビームの試料１への照射及び走査の制御を行ったりする。ビームの走査の制御は、ビーム側又は試料が固定されるステージ側、もしくはそれら両方で行うことができるが、走査速度等を考慮すると、ビーム側で制御することが望ましい。

尚、イオンビーム発生部等の構成は、特開平６－３４２６３８号公報等に記載されているような構成であってもよい。

（温度調整手段の構成）

本実施形態における温度調整手段は、先にembodiment 1でＦＩＧ．２を用いて説明したものを適用できる。

（試料の断面評価方法）

以下に、本実施形態に係る断面評価方法について述べる。なお、ここでいう試料の断面とは、評価したいの素子、材料の断面を示す。また、予め試料断面を試料の上面に設置すれば、素子や材料のある深さの表面方向の情報についても評価することが可能である。

図１１は、図１０及び図２に示す断面加工装置を用いた試料の断面評価の一手順を示すフローチャート図である。以下、図１１を参照して断面評価の手順を説明するとともに、その手順に沿った制御部９によるＳＩＭ観察のための制御及び温度制御部７ａによる試料の温度制御についても具体的に説明する。

まず、試料１を試料ステージ８の所定の位置（温度可変機構１２）に固定し（ステップＳ１０）、これを試料室３に導入した後、評価温度を設定する（ステップＳ１１）。評価温度が設定されると、温度制御部７ａにより温度可変機構１０における温度が制御されて試料１の温度がその設定された評価温度に維持される。

このときの試料１の温度は温度計９ａにて検出されており、取扱者は、不図示の表示部に表示されたその検出温度から試料１が評価温度に保たれたかどうかを確認することができる。

本実施形態においては、試料を室温より冷却した状態で加工を行うことが好ましい。また、０度以下の温度に冷却すると、試料中の水分がある場合は固化することができ、より好ましい。

このような冷却工程は、まず試料を室温以下の所定の温度に冷却し、冷却した試料を減圧雰囲気下に保持し、試料の照射面付近から発生した熱を吸収しながら収束ビームを照射することにより、照射されない部分の形状を保持したまま加工するとよい。

また、試料を冷却する際、室温状態から急速に冷却してもよい。この場合、冷却速度を４０℃／ｍｉｎ以上の速さで冷却することが好ましい。これにより、例えば温度によって分散性の変化する混合物に対しての断面形状を測定したい場合は、急冷された状態の断面を観察することができる。

該冷却工程は、減圧工程の前に行われることが好ましい。これにより、減圧による試料の蒸発を抑えることが可能となる。しかし、試料が蒸発量の少ない物質で構成されている場合、減圧と同時に冷却を行ってもよい。

冷却する工程は、対象とする試料によって異なるが、ＰＥＴ等の一般的な有機物の場合は、０℃～－２００℃、好ましくは－５０℃～－１００℃の温度範囲で冷却することが好ましい。

また、低温冷却時に加工時間、冷却時間が長くなり過ぎると、試料室内の残留ガスや、加工時に発生する物質が低温の試料に吸着してしまい、所望の加工や観察が難しくなる場合がある。このため、残留ガスや、加工時に発生する物質を吸着するトラップ手段を設け、該トラップ手段を冷却しながら加工及び情報の取得を行うことが好ましい。

本発明において、対象となる試料が有機物、特に蛋白質や他の生体物質等の熱に弱い物質や、水分を含む組成物等に好適に適用できる。特に、水分を含んだ組成物に対しては、水分を試料中に保持したまま加工することができ、好ましい。

特に、集束イオンビームを照射する場合には、減圧雰囲気下で行われる。そのため、水分を含む組成物や、揮発性の高い有機分子等に加工を施す場合、加工中に発生する熱によって水分が蒸発してしまう場合があり、本発明の温度調整手段を設ける効果は大きい。

より正確な加工及び構造評価を行うために、予め好適な加工時の保持温度を抽出する工程を備えることも好ましい。この場合、加工したい試料と等価な試料をリファレンスとして用いて、複数の設定温度において加工を行い、加工部のダメージと冷却温度の関連を調べた上で好ましい保持温度を決めるとよい。

また、一般のＦＩＢ加工装置の場合、加工後にＳＥＭ装置等の他の装置に移動して、

観察等の他の作業を行う場合が多いが、この場合、大気中に試料を出すため、一度試料温度を常温に戻した後、観察手段に移動しなければならなかった。本実施形態においては、該試料を冷却した状態で加工後、観察を行うことができ、冷却時の試料面への水滴等の付着による加工面の影響の心配がない好適な試料加工を提供できる。

試料１が評価温度に保たれたことを確認後、試料１の温度を常に確認しながら、試料１の表面のＳＩＭ観察を行う（ステップＳ１２）。このＳＩＭ観察では、制御部９によってイオンビーム発生部４によるイオンビームの照射及び試料ステージ８の移動が制御されることで、イオンビーム発生部４からのイオンビームで試料１が走査される。更に、この走査に同期して、検出器５にて２次電子（又は２次イオン：以下同様）が検出され、制御部９がその２次電子の検出信号に基づいてＳＩＭ像を不図示の表示部へ表示する。これにより、取扱者は、試料１の表面のＳＩＭ観察を行うことができる。このＳＩＭ観察は、観察用の弱いイオンビームを用いる。

次いで、試料１の表面のＳＩＭ観察によって得られた像（上記の表示部へ表示されたＳＩＭ像）から断面評価位置を精度良く決定し（ステップＳ１３）、その決定した断面評価位置を更に加工ビームでＳＩＭ観察する（ステップＳ１４）。

次いで、ＦＩＢ加工条件を設定する（ステップＳ１５）。このＦＩＢ加工条件設定では、ステップＳ１４の表面ＳＩＭ観察によって得られたＳＩＭ像上で切り出し領域及び切り出し位置を決定し、更に加速電圧、ビーム電流及びビーム径の断面加工条件を設定する。断面加工条件には、粗加工条件と仕上げ加工条件があり、この時点でそれぞれ設定される。粗加工条件は、ビームの径及びエネルギー量が仕上げ加工条件のそれより大きい。なお、切り出し領域及び切り出し位置の決定は、上記ステップＳ１２で得られる観察ビームでのＳＩＭ像上で行うことも可能であるが、精度上の問題を考慮すると、実際に加工を行うイオンビームのＳＩＭ像上で行うことがより好ましい。

ＦＩＢ加工条件が設定されると、まず、ＦＩＢ加工（粗加工）を行う（ステップＳ１６）。この粗加工では、制御部９によってイオンビーム発生部４が上記設定された粗加工条件で制御され、更に試料ステージ８の移動が制御されることで、ステップＳ１５で決定された切り出し領域及び切り出し位置に、切断に必要な量のイオンビーム

が照射される。

粗加工後、試料１の表面をＳＩＭ観察し、該ＳＩＭ観察によって得られた像（ＳＩＭ像）上で所望の位置近くまで加工されているかを確認する（ステップＳ１７）。所望の位置近くまで加工されていなかった場合は、上記のステップＳ１６及びＳ１７を繰り返す。加工された断面の表面ＳＩＭ像が極端に粗い場合も、上記のステップＳ１６及びＳ１７を繰り返すが、その際は、イオンビームの量を徐々に小さくする等の操作が加わる。ステップＳ１７における表面ＳＩＭ観察の制御は、上記のステップＳ１２の場合と同様である。

所望の位置近くまで粗加工されたことが確認されると、続いて、ＦＩＢ加工（仕上げ加工）を行う（ステップＳ１８）。この仕上げ加工では、制御部９によってイオンビーム発生部４が上記設定された仕上げ加工条件で制御され、更に試料ステージ８の移動が制御されることで、ステップＳ１６で粗加工された部分に仕上げ加工に必要な量のイオンビームが照射される。この仕上げ加工により、例えば走査型電子顕微鏡を用いた高倍率での観察を行うことができる平滑な断面を作製することができる。

次に、不図示のシャッターによってイオンビーム発生部４と検出器５を高真空に保ったまま試料室側と真空ラインを切り離した後、試料室３をリークする（ステップＳ１９）。このとき、リークに用いるガスは、評価試料材料や、評価温度によって適宜選択可能であるが、試料に水分等の付着を防ぐため、水分を取り除いたドライなガスを用いることが好ましい。例えば、窒素や不活性ガス等が用いられる。また、必要に応じて、設定温度のガスを用いることによって、試料の温度変化を最小限にすることも可能である。

更に、試料室３内のカバー７を保温部２ａごと試料１に被せ（ステップＳ２０）、カバー内部の雰囲気をガス導入部６で制御しながらカバー７を付けたまま試料１のある試料ステージを試料室から取り出す（ステップＳ２１）。

最後に、上記の様にして作製し、取り出した試料１を他の評価装置（例えばＳＥＭ）に移し、試料断面の評価を行う（ステップＳ２２）。

以上の様に、本形態の断面評価方法では、評価する試料１の温度を常に設定値に保つことができるため、ＦＩＢ加工中に試料１の状態や形態が変化することがない。よって、正確な微細構造評価を行うことができる。

以上説明した実施形態において、イオンビームによる試料の加工では、切削や研磨等の機械加工に見られるようなせん断応力、圧縮応力及び引張り応力は発生しないため、硬さや脆さの異なる材料が混合されている複合試料、空隙を持つ試料、基板上に形成した有機物の微細構造、溶媒に溶け易い試料等についてシャープな断面を作製することができる。

また、試料温度を設定値に保つことが可能なため、温度によって状態や形態が変化する材料を含む試料であっても、層の構造破壊を起こさずに、所望の設定温度で、指定した位置を直接観察することができる。

上述した各形態における断面評価方法は、ガラス等の各種基板上のポリマー構造、マイクロ粒子、液晶を含むポリマー構造、繊維状材料への粒子分散構造、温度転移材料を含む試料の所望温度の解析に対して有効である。また、イオンビーム或いは電子ビームに対してダメージを受け易い試料に対しても有効であることは言うまでもない。

尚、図１０に示した加工装置とは別に、図１２に示した装置を使用することもできる。図１２に示した加工装置は、図１０の装置にトラップ手段１６を付加したものである。トラップ手段１６の作用等については、実施形態４で説明したとおりである。

（実施形態７）

本実施形態では、図１３に示すように、試料室を試料加工を行う本体と切り離した構成の装置を示す。試料は、本体８内で加工された後、真空を保ったまま試料室３に移され、本体８と真空ラインを切り離した後、ガス導入部６から実施形態１と同様ドライガスを導入することができる。このことによって、ガス導入は、本体８に全く及ぶことがないだけでなく、試料室３を最小限の大きさにすることが可能な構成になる。従って、ガス導入部からのガス量を少なくすることが可能で、試料１の温度制御がし易くなる。

(Examples)

In the following there will be explained examples of cross sectional evaluation with the cross section evaluating apparatus of the foregoing embodiments.

(Example 1)

The present example employed the scanning electron microscope for cross sectional observation shown in Fig. 1. Temperature holding unit 2 consisted of a unit of the specimen stage with temperature controller as shown in Fig. 2, coupled with a low-temperature varying mechanism, and there was executed a cross sectional evaluation of a specimen, prepared by forming a polymer structure containing liquid crystal (two-frequency drive liquid crystal DF01XX, manufactured by Chisso Co.)(structure being obtained by mixing and polymerizing synthesized monomers HEMA, R167 and HDDA with liquid crystal) on a glass substrate, in the following procedure.

At first the specimen was fixed with carbon paste on the unit provided with the low-temperature varying mechanism, and this unit was set on specimen stage 8. After specimen stage 8 with the specimen set thereof was introduced in specimen chamber 3, the interior thereof was evacuated to a predetermined low pressure.

Then the temperature was set at -100°C, and it was confirmed that the specimen was maintained at

such evaluation temperature. Under constant confirmation of the temperature of the specimen, there was executed surfacial SEM observation of an area of the specimen including the cross section observing position. Based on the image obtained by the surfacial SEM observation, an approximately central portion of the specimen was determined as the cross section observing position.

Then the determined cross section observing position was irradiated with the ion beam to obtain an SIM image. The ion beam used in this operation was made very weak, in the observation mode. More specifically, there was employed a gallium ion source, with an acceleration voltage of 30 kV, a beam current of 20 pA and a beam diameter of about 30 nm. A cross section working portion was designated on the obtained SIM image.

Then the designated cross section working position was subjected to FIB working (crude working). More specifically, there were employed an acceleration voltage of 30 kV, a beam current of 50 nA and a beam diameter of about 300 nm to form a rectangular recess of a side of 40 μm and a depth of 30 μm in the cross section working position. The crude working was executed stepwise in small amounts under a weak condition, and the cross section of the specimen was often SEM observed in the course of

working, in order to confirm that the working proceeds close to the desired position. When the working was almost completed, the beam was switched to an electron beam and the cross section under working was so adjusted that it could be scanned by the electron beam with an angle of about 60° thereto, and an SEM observation of the cross section was executed.

After confirmation that the working proceeded to the desired position, the beam was switched to an ion beam, and the cross section working position, obtained by crude working, was further subjected to a finish working for improving the precision of the cross section working, under a weak condition similar to that in the SIM observation but with a finer beam than in the crude working. Fig. 8A schematically shows the cross section prepared by the above-mentioned FIB working, wherein a rectangular recess is formed by the irradiation of the ion beam 20, at the approximate center of the specimen 30.

Finally, the cross section of the specimen thus prepared was subjected to an SEM observation. Fig. 8B shows the mode of electron beam irradiation at such SEM observation. The cross section of the specimen 30 shown in Fig. 8A was so adjusted as to be scanned by the electron beam 21 at an angle of about 60°, and the SEM observation was executed by scanning

the cross section of the specimen 30 with the electron beam 21. The SEM observation was executed under the conditions of an acceleration voltage of 800 V and a magnification up to 50,000 x, and allows to observe the state of the liquid crystal enclosed in the polymer layer.

In this example, the cross section could be worked without deformation of the liquid crystal layer in the course of working, since the FIB working was executed while the specimen was maintained at -100°C. Also the cross section showing the liquid crystal present in the polymer could be observed since the SEM observation could be executed in the same specimen chamber while a same temperature was maintained.

(Example 2)

The present example employed the specimen stage with temperature controller shown in Fig. 5 as temperature holding unit 2, and the cross sectional evaluation of polymer particles (polystyrene) prepared on a PET substrate, was executed in the following procedure.

The temperature was set at about 10°C, and a side of the specimen was worked to form a notch of a length of about 20 μm, a width of about 10 μm and a depth of about 60 μm. In order to prevent charging phenomenon, a platinum film of a thickness of about

30 nm was deposited, prior to the FIB working, by ion beam sputtering onto the surface of the specimen. Then hexacarbonyl tungsten was introduced and an FIB irradiation was executed so as to cover the polymer particles, thereby depositing a tungsten film as a protective film. Subsequently a finish working was executed under conditions similar to those in the example 1. Fig. 9A schematically shows the cross section prepared by the FIB working, wherein a rectangular recess is formed by the irradiation of the ion beam 20, on a lateral face (corresponding to the lateral face 1a in Fig. 5) of the specimen 31.

Then an SEM of the specimen 31 in an inclined state proved that the polymer particles were closely adhered to the substrate. The SEM observation were executed under conditions of an acceleration voltage of 15 kV and a magnification up to 30,000 x.

Then the characteristic X-rays emitted from the cross section of the specimen 31 in the course of the above-mentioned SEM observation were fetched to obtain a mapping image (elementary analysis), which proved that aluminum was dispersed in polymer. Fig. 9B is a schematic view showing the irradiation of the electron beam and the emission of the characteristic X-rays at the elementary analysis. The electron beam 21 perpendicularly irradiates the cross section of the specimen 31 shown in Fig. 9A, and the

characteristic X-rays are emitted in response from the cross section of the specimen 31. The elementary analysis was executed by detecting such characteristic X-rays.

In the foregoing, there has been explained a method of evaluating the cross section of a specimen, but the present invention is not limited to such case. The present invention also includes, for example, a configuration of eliminating substances deposited on the surface, exposing a surface to be observed and observing such surface.

Also for exposing the surface, there can be employed any means capable of exposing a surface of which information is desired, and laser beam generation means can be advantageously adopted in addition to the ion beam generation means.

（実施例３）

本実施例では、図１０に示した断面加工用集束イオンビーム装置を用いた。保温部２ａとして、図２に示した温度コントローラ付き試料ステージに低温温度可変機構のついたユニットが組み込まれたものを用いて、ガラス基板上に液晶（チッソ社製二周波駆動液晶：ＤＦ０１ＸＸ）を含むポリマー構造体（重合成モノマー：ＨＥＭＡ，Ｒ１６７，ＨＤＤＡを液晶と共に混合し重合したもの）が作製された試料の断面評価を以下の手順で行った。

まず、試料を低温温度可変機構のついたユニット上にカーボンペーストで固定し、このユニットを試料ステージ８にセットした。この試料がセットされた試料ステージ８を試料室３に導入した後、試料室３内を所定の低圧力になるまで排気した。

次に、設定温度を－１００℃に設定し、試料がその評価温度に保たれたことを確認

した。試料温度を常に確認しながら試料の断面観察位置を含んだ領域について表面ＳＩＭ観察を行った。表面ＳＩＭ観察によって得られた像から試料のほぼ中央部を断面観察位置として決定した。このときのイオンビームは、観察モードのごく弱い条件で行った。具体的には、ガリウムイオン源を用い、加速電圧３０ｋＶ、ビーム電流２０ｐＡ、ビーム径約３０ｎｍとした。そして、取り込んだＳＩＭ像に対して断面加工位置を指定した。

次に、指定した断面加工位置をＦＩＢ加工（粗加工）した。具体的には、加速電圧３０ｋＶ、ビーム電流５０ｎＡ、ビーム径約３００ｎｍとして断面加工位置に４０ｍ角で、深さ３０　ｍの矩形状の凹部を形成した。この粗加工では、少しずつ段階的に弱い条件で加工するようにし、加工中は、時々、加工中の試料表面をＳＩＭ観察し、所望の位置近くまで加工されているかを確認した。ほぼ加工し終わったところで、ビームを観察用の弱いビームに切り替え、加工断面がビームに対して約６０°の角度で走査できるよう調整し、断面ＳＩＭ観察を行った。

所望の位置まで加工できていることを確認した後、更に、断面加工精度を上げるための仕上げ加工として、ＳＩＭ観察の場合と同等の弱い条件で、粗加工のときよりも細いビームで粗加工した断面加工位置を更に加工した。図８Ａは、このＦＩＢ加工により作製された断面の模式図である。試料３０のほぼ中央部に、イオンビーム２０の照射により矩形状の凹部が形成されている。途中の断面ＳＩＭ観察では、ステージを傾斜させ、観察用の弱いビームを図８Ｂに示すような角度で照射することで確認した。

次に、水分を十分取り除いたドライ窒素で試料室３の圧力をやや上昇させた後、カバー７から同様のドライ窒素をガス導入部６からわずかに導入しながら試料にカバー７をかぶせた後、試料１をＦＩＢ装置から取り出した。

最後に、上記の様にして作製した試料を試料温度に保ったままＳＥＭに導入し、断面のＳＥＭ観察を行った。

このときのＳＥＭ観察の条件は、加速電圧８００Ｖで、撮影倍率～５万倍までとした。このＳＥＭ観察により、ポリマー層の中に液晶が包まれている様子を観察することができた。

以上のように、本実施例では、試料の温度を－１００℃で維持しながらＦＩＢ加工を行ったため、加工中に液晶層がダレルこと無く、断面加工を行うことができた。また、そのままの温度を維持しながら、ＳＥＭに導入し、ＳＥＭ観察ができたため、ポリマー中に液晶が存在している様子を断面観察することができた。

（実施例４）

本実施例では、ペット基板上に作製されたポリマー粒子（ポリスチレン）の断面評価を以下の手順で行った。加工部は、試料の端部とし、評価は、ＳＥＭ観察と元素分析とした。

設定温度を約１０℃とし、試料の片側を約２０ ｍ程度の長さで１０ ｍ程度切り込む形で深さ６０ ｍ程度の加工を行った。ＦＩＢ加工前にチャージアップを防ぐため、試料表面に、イオンビームスパッタ法で膜厚１００ｎｍ程度の白金を蒸着した。他は、上記の実施例１と同様の条件で仕上げ加工まで行った。ＦＩＧ．９Ａは、このＦＩＢ加工により作製された断面の模式図である。試料３１の片側の側面部分に、イオンビーム２０の照射により矩形状の凹部が形成されている。

次に、実施例３と同様にカバーを取り付け、ＥＤＳ検出器のついたＳＥＭに導入した。試料３１を傾斜させ、ＳＥＭ観察したところ、ポリマー粒子は基板と密着していることがわかった。このときの条件は、加速電圧１５ｋＶ、倍率～３万倍程度までとした。

次に、上記ＳＥＭ観察中に試料３１の断面から放出された特性Ｘ線を取り込みマッピング像を得たところ（元素分析）、ポリマー中にアルミニウムが分散していることがわかった。ＦＩＧ．９Ｂは、その元素分析の際の電子ビームの照射及び特性Ｘ線の放出を示す模式図である。ＦＩＧ．９Ａに示した試料３１の断面に対して電子ビーム２１が垂直に照射されており、この照射に応じて試料３１の断面から特性Ｘ線が放出される。この放出された特性Ｘ線を検出することで、元素分析を行った。

尚、本実施形態では、試料の断面を評価する方法に関して説明してきたが、本発明はこれに限るものではない。例えば、表面の付着物質を取り除き、観察したい表面を露出させ、表面観察を行う構成も本願発明に含まれる。